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I, JANENE PEISKER, TEAM LEADER EXAMINATION SUPPORT AND SALES hereby certify that annexed is a true copy of the Provisional specification in connection with Application No. 2002952457 for a patent by MARS INCORPORATED as filed on 01 November 2002.



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Thirteenth day of November 2003

JANENE PEISKER
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AUSTRALIA

Patents Act 1990

ORIGINAL

PROVISIONAL SPECIFICATION

Method Of Treatment Of Vegetable Matter With Ultrasonic Energy

The invention is described in the following statement:

ULTRASONIC TREATMENT OF VEGETABLE BASED PRODUCTS

Field of the Invention

The present invention relates to the commercial processing of plant-based food materials. In particular, the invention relates to the use of low frequency ultrasonic energy to modify the viscosity of such plant based products.

Background of the Invention

The consistency or viscosity of plant-derived food materials, for example tomato based products such as pasta sauce, salsa, tomato sauce and ketchup is a key parameter in determining consumer acceptance due to its influence on visual appearance, mouth-feel and flavour release. Part of this viscosity is derived from insoluble tomato solids consisting of pectins, hemicelluloses, cellulose, proteins and lignin. These insoluble components exist in a continuous aqueous matrix of soluble pectins, organic acids, sugars and salts.

However, in most applications, the rheology of such pastes must be augmented by the use of thickening agents, such as starches (e.g. waxy maize starch) and gums such as carrageenan and guar. The disadvantage of this is that these ingredients are expensive, require specialised handling and processing and may have a deleterious effect on flavour. Therefore, it would be advantageous to be able to provide target vegetable paste rheology with minimal use of such additives.

Summary of the Invention

According to one aspect of the invention, there is provided a method of increasing the viscosity of vegetable pastes that includes treating said pastes with low frequency ultrasonic energy. Particularly advantageously, this treatment may be used to thicken or increase the viscosity of food products containing between 4°brix and 36°brix net total tomato solids including, but not limited to, tomato sauce, pasta sauce, salsa and ketchup by an ultrasonic treatment. This advantageously allows target viscosity to be achieved without necessarily using

thickening additives, as it has surprisingly been found that an ultrasonic treatment of either the tomato component of the food product or to the whole food product results in an increase in product viscosity. The exact mechanism for this viscosity increase is not known, but it is suspected that the energy released during collapse of the cavitation bubbles increases the surface area of the insoluble components, resulting in an increased interaction between the insoluble solids and the continuous aqueous matrix and also between neighboring insoluble particles.

There is a wide range of possible applications for high power ultrasound in all industries. And as a 'clean' technology, the use of ultrasound is expected to rapidly increase in importance throughout the world. The UK Department of Trade and Industry has described it as a 'key technology for the future'. The scope and potential in food processing is only now beginning to be understood and realized.

Until recently, most progress in this field was known as sonochemistry and had been carried out only at laboratory level with little work being scaled up for use on a commercial basis. Only in the last 5 years have useful advances been made in turning this laboratory-based prototype technology into fully operational commercial processes.

Ultrasound is simply sound waves at a frequency above the threshold of human hearing. It can be subdivided into three frequency ranges:

- ◆ Power ultrasound (20 - 100kHz)
- ◆ High frequency ultrasound (100kHz - 1MHz)
- ◆ Diagnostic ultrasound (1 - 10MHz)

Ultrasound achieves its chemical or mechanical effects by generating bubbles within a liquid/slurry reaction medium; a process called cavitation. The sound acts as a source of vibrational energy, which causes the molecules in the liquid to vibrate. This alternately compresses and stretches the liquid's structure to produce the bubbles. These bubbles are then subjected to those same vibrational stresses within the liquid and the bubbles eventually collapse. The conditions

within these collapsing bubbles can be dramatic, with temperatures of 5000K and pressures of up to 2000 atmospheres. As they collapse, the bubbles release energy. This is the energy that is utilized to accelerate the rate of a chemical reaction, create new reaction pathways or even generate different products from those obtained under conventional conditions.

Power ultrasound (20-100kHz) is used for most sonochemical applications, but because cavitation can be produced using sound at frequencies from within the audible range right up to higher frequencies such as 2MHz, the frequency range used for sonochemistry is expanding. But most reaction processes will operate at their optimum at 20kHz, as this is the frequency at which the maximum energy can be attained.

The use of ultrasonics in industrial processes has two main requirements; a liquid medium (even if the liquid element forms only 5% of the overall medium) and a source of high-energy vibrations (the ultrasound). The vibrational energy source is called a transducer and there are two main types; piezoelectric and magnetostrictive, the latter being less adaptable but more powerful than the former. Piezoelectric transducers are the most commonly used.

It has been observed that increasing the temperature at which the ultrasonic treatment is carried out to above the pectin melting temperature (65°C to 75°C) significantly increases the viscosity increase obtained. The pectin fraction partially binds the insoluble matrix together and it would be expected that by partially melting this pectin fraction, the insoluble matrix is more easily torn apart.

A permanent viscosity increase is obtained, but a reduction in the viscosity may be observed after the ultrasonic treatment. Two factors have been observed to effect the extent of this viscosity decrease. The first of these is the composition of the continuous aqueous matrix. It is hypothesized that the insoluble fraction is partially electrostatically bonded and that sugars and salts present in the continuous aqueous matrix partially stabilize the increased surface area structure of the insoluble component, hindering its collapse. Raising the salt and

particularly the sugar composition of the continuous aqueous phase hence reduces the viscosity reduction observed after the ultrasonic treatment.

The second factor is the application of fluid shear after the ultrasonic treatment. Fluid shear reduces viscosity, presumably by promoting contact of insoluble component with itself, allowing it to re-bond rather than to bond with the continuous aqueous matrix.

The market value of tomato based products such as pasta sauces, ketchup, salsa and tomato sauce is well known. Product viscosity is a key parameter in determining consumer acceptance due to its influence on visual appearance, mouth-feel and flavour release. The application of ultrasound to tomato based products to increase viscosity offers a range of potential market benefits including, but not limited to:

1. A reduction in the concentration of tomato solids, without a corresponding reduction in product viscosity. This reduces the cost of product formulation.
2. Improving the mouth-feel of a product and hence improving consumer preference by the presentation of a 'pulpier' product texture.
3. Increasing the viscosity of a product and hence its perceived value.
4. Viscosity has a large influence on a products flavour release. By altering the viscosity characteristics, it is possible to present a more positive flavour release. An example of this is the reduction in perceived sourness of tomato sauce following ultrasonic treatment.

Various further features and advantages of an ultrasonic treatment system for vegetable materials according to the invention are outlined below.

1. The use of low frequency ultrasound (16kHz – 100kHz) for the thickening or increasing the viscosity of food products containing between 4°brix and

36°brix net total tomato solids including, but not limited to, tomato sauce, pasta sauce, salsa and ketchup by an ultrasonic treatment.

2. Penetration of ultrasonic vibrational and cavitational energy into tomato structure/tissue to achieve increased "puffing up"/increase surface area/volume area of the cellulose surface/inner tissue/structure producing thickening of tomato based products. The use of high intensity ultrasonic radial or focused sonotrodes into the liquid/food flow stream emitting high shear energy waves into the structure/tissue/body of the food product to thicken tomato based products unlike existing ultrasonic patents which use the concept of transducers clamped/bolted/welded to the outside of steel vessels/chambers. This effect creates low energy, low efficiency standing/stationary waves in the liquid/food allowing for only outer surface treatment in a batch set-up. There is no penetration of standing wave energy into the food structure to achieve thickening. By way of example, the energy intensity of associated with standing/stationary waves is only in the region 0.001 Watt/cm³ which is insufficient to achieve penetration in the food product. In contrast, the introduction of high intensity transducers/sonotrodes to the liquid stream produces intensities of between 1 – 1000 W/cm³ allowing for penetration in the food product on a batch and continuous flow basis.
3. Provide ultrasonic micro-streaming to increase the "puffing up"/increase surface area/volume area of the cellulose surface/inner tissue/structure in tomato based products. This is achieved by increasing the penetration of energy/shear into the tomato based product which may be retarded under conventional conditions due to hydrophobicity of the plant surface layer. This concept is only possible by the development and introduction of transducer/sonotrodes which are immersed in liquid/product flow using high intensity radial wave propagation or focused systems. The use of high intensity ultrasonic radial waves or focused energy produces microstreaming effects/cavitation effects on the surface or into the structure/tissue/body of the food product on a batch or continuous flow

treatment set-up. In contrast existing ultrasonic patents which use the concept of transducers clamped/bolted/welded to the outside of steel vessels/chambers. This effect creates low energy, low efficiency standing/stationary waves in the liquid/food which does not produce microstreaming effects to enhance penetration and puffing of cellulose structure. By way of example, the energy intensity associated with standing/stationary waves is only in the region 0.001 Watt/cm³ which is insufficient to achieve penetration microstreaming effects in the food product on a batch or continuous flow basis and insufficient to achieve enhanced energy/shear penetration and "puffing" of cellulose structure/tissue. In contrast, the introduction of high intensity transducers/sonotrodes to the liquid stream produces intensities of between 1 – 1000 W/cm³ allowing for high velocity microstreaming effects (780 km/hr), enhanced energy/shear penetration and "puffing up" of cellulose, increased surface area/volume of the tissue/structure on a batch and continuous flow basis.

4. Increased performance of the thickening process for tomato based products and thickening efficiency by the introduction of specific designed radial sonotrodes for different types of tomato based products. The greater the organic load, type of plant surface/tissue/structure will determine the type of sonotrode design. The design of a specific sonotrode will allow for greater penetration of the ultrasonic wave/cavitation energy, better coupling, impedance matching of energy to product and improved energy efficiency resulting in greater thickening of tomato based products. By way of example, a tomato based product with low organic loading or products with a strong absorption factor of ultrasonic energy would use a cascade design radial sonotrode. In contrast, a process with a high organic load or a low ultrasonic absorption coefficient would use a different radial sonotrode. This sonotrode would have an increased diameter to length (wavelength) factor /ratio producing ultrasonic waves which have a greater distance of propagation and have a greater capacity to penetrate through a product stream with a high organic loading or a product with a low

ultrasonic absorption coefficient. Existing patent designs using transducers welded/bolted to the outside of vessels/chambers/tubes were not designed for specific types of organic load or products with different ultrasonic absorption characteristics.

5. Increased performance of the thickening process of tomato based products by the introduction of an automatic frequency scanning system for different types of tomato based products. The greater the organic load (concentration, viscosity), type of plant surface/tissue/structure will determine the resonance frequency of that system/tomato based product. The ultrasonic resonance frequency is the frequency at which the ultrasonic unit will deliver the greatest energy efficiency. The ultrasonic system will lock onto the resonance frequency of a specific tomato based product and then re-scan for the new resonance frequency every 0.001 second throughout the treatment process. If the ultrasonic unit did not have the resonance frequency tracking system for different products, then if there was as little as 10Hz variation from the resonance frequency, this would result in a drop in energy efficiency in the order of 10 – 40%. This would have a significant reduced effect on the thickening process of tomato based products. By way of example , the resonance frequency of lettuce is 20,350 Hz where as tomato sauce will have a resonance frequency of 20,210Hz. Existing patent designs using transducers welded/bolted to the outside of vessels/chambers/tubes were not designed with an automatic resonance frequency tracking system for specific types of tomato based products so products could not be processed at the correct resonance frequency and maximum power efficiency.
6. The use of high power and high amplitude ultrasound (0.01W/cm² – 1000W/cm² and 1 micron displacement – 500 micron displacement) for the thickening of tomato based products. Existing patents were unable to produce this kind of energy level, therefore thickening of tomato based on a continuous flow food product stream would not be possible.

7. The use of low frequency/high intensity ultrasonic focused energy from a specially designed sonotrode or transducer for the thickening of tomato based products. The focused sonotrodes are immersed in the continuous flow stream rather than bolted to the outer walls of the vessel. The immersed sonotrodes produce high velocity microstreaming effects enhancing the penetration of solvent into the food product and enhance thickening of tomato based products.
8. With reference to items 1 & 8 above, the sonotrodes/transducers can be retrofitted into a bath, trough, vessel, chamber, pipe, flow cell containing the tomato based products for the thickening process. The sonotrodes are in contact with the liquid/product stream producing greater energy density, microstreaming, cavitation energy, energy efficiency and thickening yield.
9. With reference to items 3, 7 and 8 above, the sonotrode could be made of titanium, aluminum, steel, hastalloy, ceramic, glass.
10. With reference to items 1 – 9 above, ultrasonic performance enhancement at both high and low temperature of the solvent for thickening of tomato based products on a batch or continuous flow basis. The process can be carried out in a temperature range of between 10°C and 100°C. Ideally, the process will be carried out in a temperature range of between 70 and 80°C.
11. With reference to items 1 – 10 above, ultrasonic performance enhancement at pressures greater than 0 bar (0.1 bar – 10 bar pressure) for thickening of tomato based products on a batch or continuous flow basis. Increase in pressure causes an increase in load on the transducer and this produces a greater draw of energy from the PZT crystal (PIEZO CERAMIC CRYSTAL) inside the transducer. The net result is an improved energy efficiency.

12. With reference to items 1 – 11 above, ultrasonic performance enhancement for the thickening of tomato based products with very low viscosity/high water content as well as high viscosity products with low water composition. The use of high power, energy efficient focused and radial systems producing high velocity microstreaming and radial wave propagation which enables the penetration of high shear cavitation energy through the hydrophobic surface and into the tissue/structure of the tomato products.

13. Enhanced disintegration of tissue, particle size breakdown of tissue and rupture of cells using ultrasonic vibration, cavitation and microstreaming to increase the thickening of tomato based products. Disintegration of cellulose tissue and cell rupture inside a food product could not be achieved on a continuous flow basis using energy levels (0.001w/cm³) associated with existing patent designs for thickening.

By way of example, an outline of some of the equipment that may be used to carry the invention into effect includes:

A power source, a transducer converting electric energy into mechanical vibrational energy, which is transmitted into the tomato based products system by a sonotrode. The sonotrode can provide either radial wave, stationary wave or focused emission, depending on the application (see designs). The sonotrode material can be made of titanium (preferentially) or ceramic, steel, hastalloy, glass. The transducer system could be PZT (piezo ceramic transducer), Terfenol-D magnetostrictive transducer or a Nickel/Iron/Vanadium magnetostrictive material. Application of ultrasonics range 20kHz to 100kHz), intensity 400W/cm² – 1000W/cm²) and amplitude (1 micron displacement – 500 micron displacement). The transducers/power supply can have an individual power ranging from 100 Watts – 8000 Watts. The power supplies have automatic resonance frequency tracking so that when the equipment is running during the thickening process, the unit is also always scanning the new resonance frequency (relates to maximum power output) due to changes in the liquid stream.

The sonotrodes/transducers can be mounted or retrofitted to tanks, vessels (round, square, oval), troughs, pipes, flow-cells containing the tomato based products.

Radial sonotrodes may be fitted longitudinally or laterally within an open tank, trough or flume. Reflector shields may be positioned at the base to reflect and/or focus ultrasonic energy into the product flow path, which may be at the solvent surface or immersed below the surface.

Radial sonotrodes may be fitted in closed flow through cells, sonotrode design to enhance thickening efficiency of the solvent, inclusion of reflector shields to improve efficiency.

Focused sonotrodes may be fitted in flow through cell where the produce and solvent flows either directly into the face or across the face of the sonotrode. Residence time can be controlled by regulating the micro-streaming flow from the sonotrode the solvent flow rate (from more than one direction) and vessel design.

DATED this 1st day of November 2002

MARS INCORPORATED

WATERMARK PATENT & TRADE MARK ATTORNEYS

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